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Superconducting properties of small-filament Th-Nb eutectic composite

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We have measured the electrical resistance, dc magnetization, and thermal conductivity of a drawn Th-Nb eutectic composite consisting of small Nb filaments in a Th matrix. A weak heat treatment can be used to destroy the filamentary geometry. We find an upward curvature in H_{c2} which is greatly reduced when the filaments are destroyed, suggesting that its origin may lie in the small-scale quasi-one-dimensional nature of the material. However, because of the proximity effect the mechanism of a decoupling transition does not seem applicable to this material. The transition temperature of the unannealed composite is higher than would be expected on the basis of the proximity effect. This result has been observed in other composites and may be due to some type of coupling between the filaments.

I. INTRODUCTION

There has been much recent interest in the superconducting properties of filamentary or quasi-one-dimensional materials such as $(SN)_x$,[1-5] $Hg_{3-\delta}AsF_6$,[6-9] or $NbSe_3$.[10] Studies of these materials have led to the observation of several interesting effects including fluctuation conductivity above T_c,[5] upward curvature in H_{c2},[1,4] anisotropic Meissner effects[3,4,8] and critical fields,[1,10] and possible dimensional crossovers.[11] Despite the experimental progress there is no general understanding of these phenomena. In many cases it is difficult to determine whether the observed phenomena are due to the filamentary morphology of the material, whether they are intrinsic to the material, or if their origin lies with the ever present sample imperfections and inhomogeneities.

We report here on the superconducting properties of a composite material consisting of aligned Nb filaments in a Th matrix. The composite is produced by directional solidification from the melt at the eutectic composition followed by drawing of the wire to produce samples with Nb filaments having a thickness of less than the Nb coherence length. A weak heat treatment causes a coarsening of the composite, breaking the filaments up into a collection of discontinuous particles. By measuring both the annealed and unannealed samples it is possible to some degree to isolate the effects of the filamentary morphology. In particular, we find the small scale filaments important for the observation of upward curvature in the upper critical field H_{c2}.

For a material which is less than 10 wt. % Nb the superconducting properties of the composite are surprisingly strong. The entire sample exhibits a Meissner effect and can exclude fairly large fields. The transition temperature is near that of bulk Nb even for samples with Nb thicknesses much less than a coherence length—much higher than predicted by the proximity effect. There is as yet no generally satisfactory explanation for these results but we note that unexpectedly high transition temperatures have been observed in other composite materials[12,13] as well. Our measurements are consistent with the idea that some type of coupling between the superconducting filaments can increase the transition temperature of a composite.[14]

A description of the samples is contained in Sec. II. Sections III and IV describe the resistive and magnetic measurements. The transition temperature is discussed in Sec. V and the thermal-conductivity measurements in Sec. VI.

II. SAMPLES

The samples were formed by directionally solidifying a mixture of pure Th and Nb in a floating-zone refiner.[15] The metals were mixed at the eutectic concentration (7.07 wt. % Nb) and the resulting composite contained a triangular lattice of aligned cylindrical Nb filaments in a Th matrix. The low mutual solubility of the two metals (0.1 wt. % or less[16]) and the formation from the melt assures us of two clean phases in good electrical contact (i.e., no oxide barrier). The Nb filament diameters are uniform for a given sample and are determined by the velocity of the floating zone. Composites with filaments as small as 1000 Å have been made.

The composite wire, which has an overall diameter of 3.2 mm after soldification, is then drawn down to a diameter in the range 0.2 to 1.0 mm. Electron micrographs show that the drawing makes the Nb ribbonlike, changing the cross section from circular to nearly rectangular with sides in the ratio of roughly 6 to 1. We refer to the length of the smaller dimension as the thickness t of the Nb filaments. Samples used in this study covered the range from $t \sim 30$ to 210 Å. Thus the samples have at least one dimension which is smaller than the coherence length of pure Nb (380 Å).[17] Although the drawn filaments no longer form a regular lattice, electron micrographs show that the filaments still remain uniformly distributed throughout the sample. The filaments tend to align themselves so that the long dimension of the rectangular cross sections are roughly parallel.

The resulting wire is in some ways similar to another composite wire consisting of a copper alloy containing Nb particles which is drawn down by a large factor.[18,19] The Nb particles are randomly

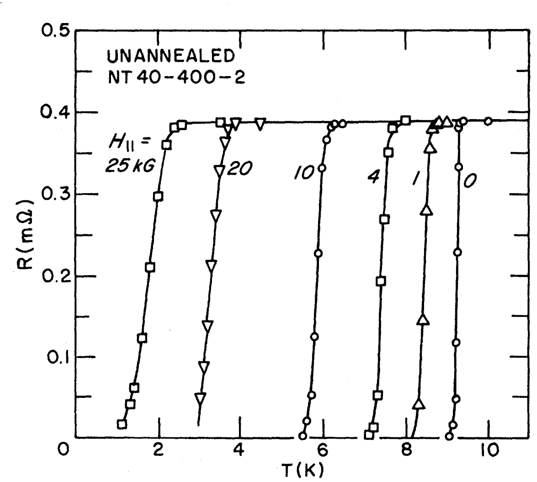

FIG. 1. Resistance vs temperature in several magnetic fields for an unannealed Th-Nb sample with $t = 145$ Å. Direction of the current and magnetic field are parallel to the filaments.

dispersed in the copper and the drawing causes them to form discontinuous filaments and leads to a percolation conduction when the Nb is superconducting. By contrast, the filaments in the eutectic composite are continuous as well as smaller in cross section making them more closely resemble a quasi-one-dimensional material. As will be seen in the next section the copper alloy and the eutectic composite have very different electrical properties.

Measurements were also made on samples which had been annealed for one hour at 850 or 900 °C. Although this is well below the 1435 °C melting temperature[16] for the composite it was sufficient to destroy the filamentary morphology. The coarsening resulted in a dispersion of discontinuous Nb particles of $\sim$2000-Å diameter and appeared in electron micrographs to be independent of the diameter of the original filaments.

III. ELECTRICAL RESISTANCE

Four terminal electrical resistance measurements were made over a (1 − 3)-cm length of sample using copper clamps to make electrical contact. The sample could be mounted in a helium cryostat with the filaments either parallel or perpendicular to the field generated by an external superconducting magnet. The normal-state resistance for all samples is temperature independent below $\sim$10 K and corresponds to resistivities of 1.0 − 1.8 $\mu\Omega$ cm for the unannealed (as drawn) samples and 0.4 − 1.0 $\mu\Omega$ cm for the annealed samples.

We discuss the unannealed samples first. A typical resistive measurement is shown in Fig. 1. There is a single resistive transition at a temperature T_{cR} (defined by the midpoint of the transition which is $\sim$9 K for all samples measured) with a width $\sim$0.3 K which increases with increasing field. The single transition is also confirmed in measurements made with a superconducting quantum interference device (SQUID) voltmeter in which the resistance is followed down more than 5 orders of magnitude below the normal-state resistance. The curves exhibit only a weak current dependence. This is quite different from the resistance-versus-temperature curves of the drawn copper alloy[18,19] in which there is a plateau in the resistance between the superconducting transition of the Nb inclusions and the percolation threshold at which the proximity effect can complete a zero-resistance path. The filaments of the eutectic composite are continuous so there should be a zero-resistance path at all temperatures at which the Nb is superconducting.

Even most of the annealed samples show only a single transition presumably because of the strong proximity effect in the Th. A few, however, do show a slight plateau region shown in Fig. 2 but this probably does not arise from the same mechanism as in the copper alloy. Only a few samples show this behavior with no consistent pattern to its occurrence. Moreover, the plateau occurs at a fraction of the normal-state resistivity which differed from sample to sample and did not correspond to a level which might be expected from superconducting Nb in a normal Th matrix. It seems likely that the plateau is due instead to some imperfection or inhomogeneity in the sample.

We determine $H_{c2}(T)$ from the resistance curves as being the field such that $R(T)$ is one-half the normal-state value. Typical values for the upper critical field, both parallel and perpendicular to the axis of the filaments for the unannealed samples are shown in Fig. 3. The error bars shown on one of the curves correspond to the temperatures at which the resistances are 20% and 80% of their normal-state values.

The upper critical field shows an upward curvature similar to that observed in the fibrous material $(Sn)_x$ (Refs. 1 and 4) and in several layered compounds.[20–22] For filamentary materials Turkevich and Klemm[23] predict an upward curvature by the following mechanism. Near T_c the Ginzburg-Landau coherence length is large enough to couple the filaments together so that the material acts like an anisotropic three-dimensional superconductor. At lower temperatures the coherence length is reduced allowing the filaments to decouple. At this point the material acts like a collection of isolated superconducting filaments which have a divergent

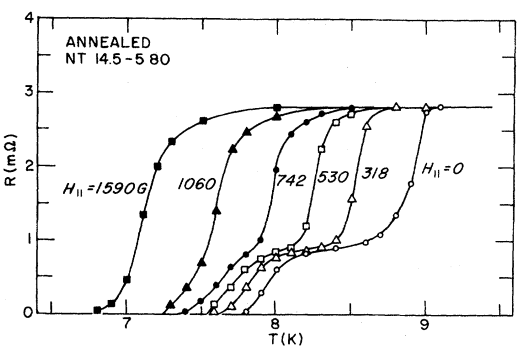

FIG. 2. Resistance vs temperature in several magnetic fields for an annealed sample of Th-Nb. Only a few of the annealed samples showed a plateau in zero field.

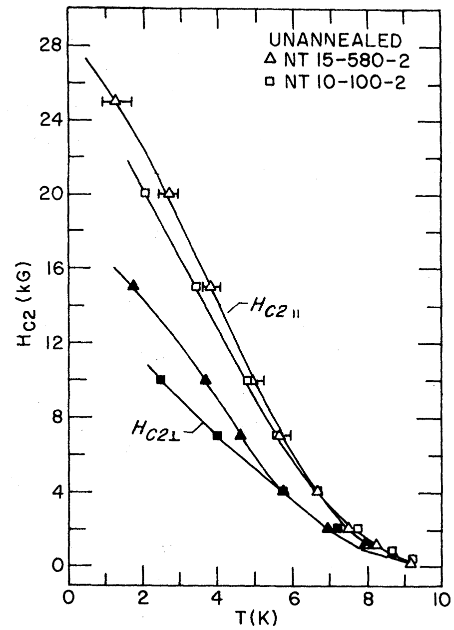

FIG. 3. Upper critical field vs temperature for two unannealed samples for both the case of the field parallel and the case of the field perpendicular to the filaments. Error bars show the points of 20% and 80% of the normal-state resistance for sample NT 15-580-2. Sample NT 15-580-2 has $t = 210$ Å and sample NT 10-100-2 has $t = 36$ Å.

H_{c2} for very small filaments. Nevertheless it has not been possible to calculate the full temperature dependence of H_{c2} for $(SN)_x$ using a consistent set of parameters and as a result the origin of the upward curvature is uncertain.

It is not at all obvious that the mechanism described by Turkevich and Klemm should apply to the Th-Nb composite. Unlike the superconducting filaments in an inert matrix pictured in their model, the filaments of the composite are surrounded by Th which enhances the coupling of the filaments through the proximity effect. The relevant length for this coupling in the Th is the pair potential decay length[24] K_N^{-1} which in the short mean-free-path limit is given by

$$K_N^{-1} = \left[\frac{\hbar v_F l}{6\pi k (T - T_{cn})} \right]^{1/2} \cong \frac{1100\ \text{Å}}{(T - T_{cn})^{1/2}} \tag{1}$$

for Th with a resistivity of $\sim 1\ \mu\Omega\,\text{cm}$. This length is comparable to the distance between filaments although it has been suggested that K_N^{-1} is reduced by magnetic fields.[25] This decay length also *increases* with decreasing temperature—just the opposite of the Ginzburg-Landau coherence length—so the filaments should be more strongly coupled at lower temperatures. The magnetization measurements of the next section confirm the enhanced low-temperature coupling of the filaments at least in low fields. But there is evidence of coupling in large fields as well. The weak coupling between filaments in most quasi-one-dimensional materials leads to a very large anisotropy in H_{c2} [e.g., $H_{c2\parallel}(0)/H_{c2\perp}(0)\sim 30$ (Ref. 1) in $(SN)_x$]. But for the Th-Nb composite $H_{c2\parallel}(0)/H_{c2\perp}(0)$ is much lower and is within the range $1.4-2$ for all samples. In addition, H_{c2} of the composite shows very little dependence on the diameter of the filaments in contrast to what would be expected for decoupled filaments.

If the coupling between filaments does make the material an anisotropic three-dimensional superconductor we can apply the standard Ginzburg-Landau theory to determine the relevant coherence length.[26] Making a rough extrapolation to $T=0$ we take the average values $H_{c2\parallel}(0)=28$ kG and $H_{c2\perp}(0)=16$ kG which lead to $\xi_\perp=(\phi_0/2\pi H_{c2\parallel})^{1/2}=110$ Å and $\xi_\parallel=\xi_\perp(H_{c2\parallel}/H_{c2\perp})=190$ Å. Many samples thus had Nb thickness such that $t<\xi_\perp,\xi_\parallel$. If we assume that these coherence lengths pertain to the Nb and use them to calculate a resistivity[27] we find $\rho_\perp=1.2$ $\mu\Omega$ cm and $\rho_\parallel=3.6$ $\mu\Omega$ cm which are reasonable values being quite similar to that measured for the composite (which is mostly Th). So the measured $H_{c2}(0)$ is close to what would be calculated for bulk Nb having a resistivity similar to that of the composite.

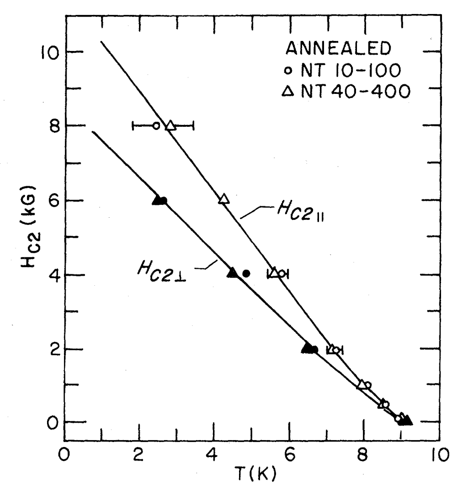

FIG. 4. Upper critical field vs temperature for two annealed samples for both the case of the field parallel and the case of the field perpendicular to the long dimension of the wire. Error bars show the points of 20% and 80% of the normal-state resistance for sample NT 10-100.

The values of the H_{c2} for the annealed samples are shown in Fig. 4. In contrast to the unannealed samples there is little evidence for upward curvature. The H_{c2} values are nearly linear in T_c-T. This suggests that whatever the cause of the upward curvature it is related to the small-scale filamentary morphology.

The H_{c2} values for these annealed samples are considerably smaller than for the unannealed case which is consistent with increased conductivity on annealing. The low-temperature values of $H_{c2\parallel}(0)=11.5$ kG and $H_{c2\perp}(0)=8.5$ kG correspond to $\xi_\perp=170$ Å and $\xi_\parallel=230$ Å which are much smaller than the Nb-particle size. The anisotropy is less $[H_{c2\parallel}(0)/H_{c2\perp}(0)\sim 1.3]$ than that in the unannealed samples and presumably reflects the strains which were not relaxed by the weak heat treatment. There is less variation in H_{c2} among the annealed samples as expected from the small variation in structure seen in electron micrographs.

IV. MAGNETIZATION MEASUREMENTS

To determine the fraction of the sample that was actually superconducting, measurements of the dc magnetization were made using a SQUID magnetometer. The measurements were dc to avoid the heating and short skin-depth effect of ac measurements in the high-conductivity mixed state. A sample would be greased to a copper sample holder and placed in one end of a gradiometer coil which was connected to the SQUID. A magnetic field could be generated with a superconducting coil and trapped in a Nb cylinder in which the gradiometer coil and sample resided. The sample was placed with the filaments parallel to the applied field. The helium Dewar was contained within a magnetic shield which reduced the residual field below 5 mG.

To make a measurement the sample is cooled to the lowest temperature and a magnetic field generated with the superconducting coil. The gradiometer coil and Nb cylinder are heated above their transition temperatures and then allowed to cool, trapping flux in the cylinder. As the temperature of the sample is increased the change in feedback current to the SQUID is proportional to the change in magnetization M of the sample. The volume from which the field was excluded (and hence the magnetization) was calibrated by measuring a sample of Pb wire similar in size and shape to the eutectic composite.

Typical results are shown for an unannealed sample in Fig. 5. The data show a Meissner effect at low temperatures which extends not just over the small fraction of the sample which is Nb but over the Th as well. Even in the lowest fields used, the exclusion of magnetic flux does not occur at T_{cR} but at a somewhat lower temperature. If we define T_{cM} as the midpoint of the magnetic transition we find it usually occurs at a temperature 0.5–3 K below the resistive transition. In this intermediate-temperature range ($T_{cR} > T > T_{cM}$) it does not seem likely that the combination of nearly zero resistance and magnetization is due to an irregularly large thread of Nb acting as an electrical short. There was little current dependence (as might be expected for a single thread of superconductor) and such a thread has not been detected in numerous electron micrographs of samples in which the Th has been chemically etched away.

Data on other quasi-one-dimensional materials show evidence for different resistive and magnetic transitions as well. In $(SN)_x$ the beginning of flux exclusion is usually found at a lower temperature [0.23 K (Ref. 2) and 0.25 K (Ref. 3)] than the transition to zero resistance [0.255 K (Ref. 1) and 0.29 K (Ref. 5)]. In $Hg_{3-\delta}AsF_6$ the resistance (at least in one direction[9]) drops to zero near 4 K. Some flux exclusion is observed below this temperature but it is very small and field dependent.[8] A full Meissner state is not observed until a much lower temperature of 0.43 K.[9]

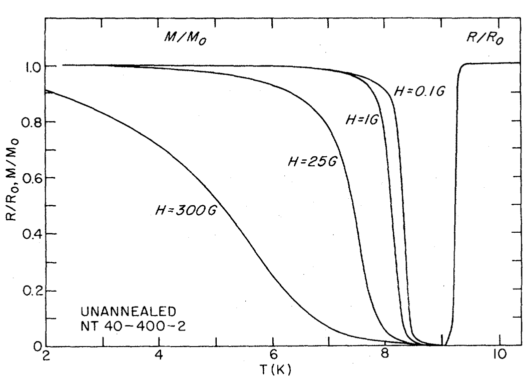

FIG. 5. Normalized resistance and dc magnetization vs temperature for an unannealed sample of Th-Nb having $t = 145$ Å. Magnetization measurements were made for several values of the field applied parallel to the filaments.

A small, field-dependent magnetization at T_{cR} may also be occurring in Th-Nb but our resolution may be insufficient to detect it. We note that the magnetization is field dependent down to the smallest fields used (0.1 G) with T_{cM} increasing for smaller fields. It may be that even very weak fields can suppress the magnetic transition. This effect does not appear to be related to the filamentary morphology of the composite for it also occurs in the annealed composite (Fig. 6).

We interpret these two transitions in a manner similar to that which has been proposed for granular superconducting film of NbN (Ref. 28) and Al.[29] For these films it is suggested that there is an initial transition when the individual grains go superconducting and a second transition at a lower temperature at which coupling of the phases of adjacent grains takes place. Analogously, in Th-Nb we take T_{cR} to be the onset of superconductivity in the Nb and T_{cM} as the transition for phase coupling between Nb regions. In this picture the superconducting regions become more strongly coupled at lower temperatures—precisely the opposite of the decoupling transition discussed in the previous section. This would be consistent with the expected behavior of the proximity effect in the Th.

The curves of Figs. 5 and 6 can be used to define a lower critical field H^*_{c1}. In Fig. 7 we show the values of H^*_{c1} determined by extrapolating[30] the M-vs-T curve along the region of steepest slope to the point where M has its full Meissner-state value M_0. In practice this turns out to be very near the field at which $M \sim 0.8 M_0$. Near T_{cM} the fields are small but they become quite large at lower temperatures; much greater in fact than the 159-G critical field[31] of Th at $T = 0$. These low temperature values are

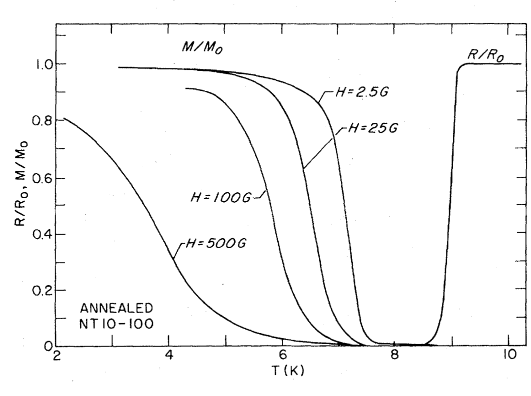

FIG. 6. Normalized resistance and dc magnetization vs temperature for an annealed Th-Nb sample.

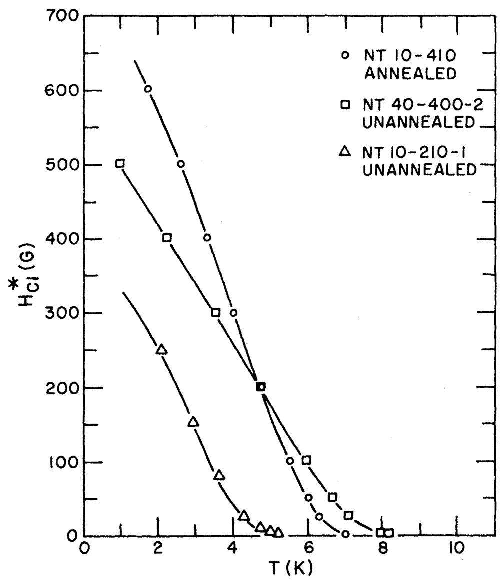

FIG. 7. Field H^*_{c1} vs temperature for several samples of Th-Nb. Sample NT 10-210-1 has $t=76$ Å and sample NT 40-400-2 has $t=145$ Å.

much too large to be a true lower critical field H_{c1}. They are larger than the calculated thermodynamic critical field for the composite and larger than the $H_{c1}(0)$ which would be calculated for bulk Nb having the same upper critical field as the composite [using $H_{c2\parallel}(0)$ of the composite as H_{c2} for bulk Nb would give the bulk Nb a $\kappa \sim 10$ and $H_{c1} \sim 300$ G]. Instead we believe that the large H^*_{c1} values reflect the strong pinning forces in this homogeneous material. This explanation is consistent with the irreversibility of the magnetization curves and the large critical currents found in the composites.[18]

V. TRANSITION TEMPERATURE

A superconductor of thickness less than a few coherence lengths is expected to have a reduced transition temperature when it is in contact with a normal metal. For the annealed samples, the Nb regions are large and little reduction in T_c is expected but for the unannealed samples this is not the case. For simple film geometries it is possible to calculate[32,33] the T_c as a function of the various film thicknesses but for the geometry of the unannealed composite an exact calculation would be quite difficult. However, the thinnest samples of the unannealed composite should be in the Cooper limit (thickness small compared to the coherence length) so that T_c should not be too dependent on the geometry and hence easier to estimate. de Gennes[24] calculates that in this limit, T_c can be determined by averaging $N(0)V$ (and using the expression $T_c = 0.85\Theta_D e^{-1/N(0)V}$) over the composite and weighting the average by the density of states at the Fermi surface $N(0)$. With an order of magnitude more Th in the composite, even weighting with $N(0)$ (which tends to make the Nb more significant), the T_c should only be ~ 2.4 K; only slightly above that of bulk Th [for which $T_c = 1.38$ (Ref. 31)]. Entin-Wohlman and Bar-Sagi[34] suggest that there should be an additional weighting factor of the inverse coherence length which again tends to increase the effect of the Nb because of its shorter coherence length. This would raise the calculated T_c to ~ 4.1 K. Nevertheless, all measured unannealed samples, even those with $t \sim 30$ Å have a resistive transition at around 9 K and show a complete Meissner effect well above 4 K. With such a large amount of Th surrounding each Nb filament it seems unlikely that a better calculation, perhaps taking into account the composite geometry, could bring the calculated T_c more in line with the experimental data.

Unexpectedly high T_c's have also been observed in other eutectic composite systems. Matthias *et al.*[12] have investigated a eutectic composite of Ir and YIr_2 which has a T_c of 2.7–3.7 K which is well above the transition temperature of its components [$T_c(\mathrm{Ir}) = 0.1$ K, $T_c(\mathrm{YIr_2}) < 0.02$ K]. They suggest that T_c is enhanced due to a lattice softening which is indicated by a drop in the Debye temperature from 420 K for pure Ir to 175 K for the eutectic composite. Heat-capacity measurements on the undrawn Th-Nb composite[35] show no significant change in the Debye temperature but these composites had much larger Nb filaments (~ 5000-Å diameter) than the samples studied here and hence does not rule out the possibility of a lattice softening.

Granquist and Claeson[13] have mimicked a eutectic composite by evaporating alternating layers of a superconductor (either Sn or In) and normal-metal (Ag) layers at low temperatures to prevent interdiffusion. Surprisingly, T_c increased with increasing number of NS bilayers. A stack of $2m$ films (m of Sn or In, m of Ag) had a T_c greater than one consisting of $2m-2$ films ($m-1$ of Sn or In, $m-1$ of Ag). After a small number of bilayers have been added the T_c saturates at a value near or slightly greater than the T_c of the single superconducting film. Their results were interpreted in terms of a phenomenological theory of Kulik[14] which predicts the transition temperature for a set of interacting layers of superconducting material. It is found that an interaction of the Josephson type cannot cause an increase in the transition temperature but that an "electron correlation" between layers can. A microscopic picture of the postulated interaction is not provided but it is of the form of a Coulomb-type interaction in which an electron in one layer is scattered by an electron in a different layer with no transfer of electrons between layers. The T_c of the evaporated films is consistent with this calculation but it is by no means conclusive.

We speculate that whatever mechanism is responsible for the enhanced T_c of the evaporated films may also be operating in the unannealed Th-Nb composite. The distance between Nb filaments is of the same order of magnitude as the 100 Å of Ag between the superconducting films; and the transition temperature of Th-Nb is quite close to the T_c of Nb alone, as with the saturated T_c of the bilayers.

VI. THERMAL CONDUCTIVITY

The thermal conductivity κ of the unannealed composite was determined as a further measure of superconductivity in the Th. The measurements were made in a direction parallel to the axes of the Nb filament. The component of the thermal conductivity due to phonons should be negligible[36] and in the normal state (i.e., in a large field), κ_N was proportional to T below ~ 10 K as expected for the electronic component. In the superconducting state the conductivity was somewhat lower and began decreasing rapidly at the lowest temperatures. The ratio κ_s/κ_N is shown in Fig. 8. A small correction has been made which is less than 10% at all temperatures to subtract off the estimated conduction due to Nb. There is presently no full theory of the thermal conductivity in the proximity effect regime so we make the crude approximation[37] that the Th behaves like a homogeneous superconductor. This approximation gains validity from the small dimensions of the Th. Bardeen, Rickaysen, and Tewordt[38] have calculated the thermal conductivity of a homogeneous superconductor as a function of the energy gap Δ. This calculation for an ordinary BCS superconductor ($2\Delta_0/kT_c=3.53$) is shown as the dashed line in Fig. 8 and is much lower than the actual data. The solid line is the same calculation with a low-temperature energy gap $\Delta_0/k=4$ K. This calculation omits any effects due to Andreev scattering[39] but because two samples with Nb dimensions differing by a factor of 2 give similar results this omission seems justified. This value of Δ_0 is about 50% greater than the low-temperature value for bulk Th but it is certainly much smaller than would be the case if some type of lattice softening had caused the composite to behave as a 9-K BCS superconductor.

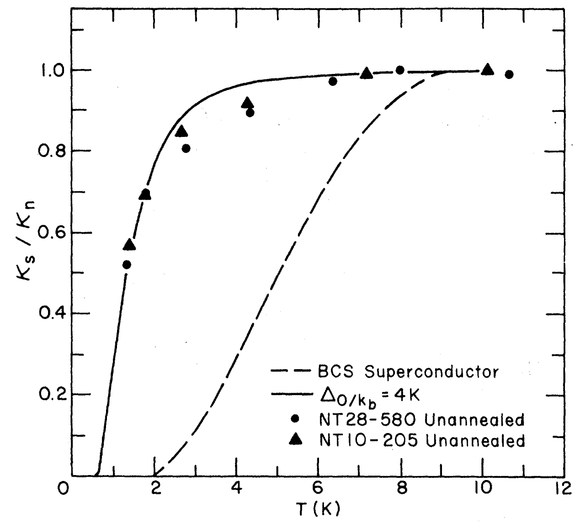

FIG. 8. Ratio of the thermal conductivity in the superconducting state to that in the normal state for two unannealed Th-Nb samples. Dashed line is the Bardeen-Rickaysen-Tewordt calculation for an ordinary BCS superconductor ($2\Delta_0/kT_c=3.53$). Solid line is a similar calculation for a smaller energy gap. Sample NT 28-580 has $t=210$ Å and sample NT 10-205 has $t=74$ Å.

VII. CONCLUSION

We have studied several of the superconducting properties of a drawn Th-Nb eutectic composite. The composite shows several characteristics which are similar to other quasi-one-dimensional materials yet is somewhat better characterized since the filaments can be seen in electron-microscope pictures. The filamentary morphology can be destroyed by a weak heat treatment. We find an upward curvature in H_{c2} only for the unannealed sample suggesting that it is related to the small-scale filamentary structure. The proximity effect of the Th (and the resulting small anisotropy in H_{c2}) would seem to point against the much discussed decoupling transition.

The high transition temperature of the unannealed composite cannot be explained solely by the conventional proximity effect. The existence of millions of filaments in each sample is consistent with the data of Granquist and Claeson and the interpretation that some type of coupling between filaments is responsible for the enhanced T_c. The details of such a mechanism are still not known.

The thermal-conductivity data show that the high T_c is not the result of a mechanism which simply increases the electron-phonon interaction since that would increase both T_c and Δ_0. Hence a lattice softening is not consistent with this data.

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